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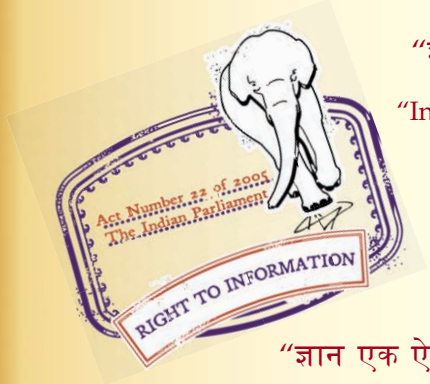
IS 3088 (1967): Method for assaying of fine grade palladium [MTD 10: Precious Metals]



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IS : 3088 - 1967
(Reaffirmed 2003)

Indian Standard

METHOD FOR ASSAYING OF FINE GRADE PALLADIUM

UDC 669·234 : 543 : 2

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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Indian Standard

METHOD FOR ASSAYING OF FINE GRADE PALLADIUM

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Indian Standard

METHOD FOR ASSAYING OF FINE GRADE PALLADIUM

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 June 1967, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Palladium is finding increasing use in the industry. In order to determine the purity of palladium, it is necessary that a standard method is followed. With this in view, a method of assaying has been given in this standard which shall be found useful both for routine and reference purposes. Palladium in its refined form is likely to contain traces of silver, gold and platinum. While preparing this standard, the presence of these elements has been taken into consideration.

0.3 In preparing this standard, the Sectional Committee took into consideration the manufacturing and trade practices followed in the country in this field.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the method for determining the purity of fine grade palladium as laid down in IS : 3096-1965†.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960‡), shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Rules for rounding off numerical values (*revised*).

†Specification for fine grade palladium.

‡Specification for water, distilled quality (*revised*).

3. DETERMINATION OF PALLADIUM CONTENT IN FINE GRADE PALLADIUM

3.1 Outline of the Method — After the separation of impurities like silver, gold and platinum, palladium is precipitated as dimethylglyoximate, filtered, dried and weighed.

3.2 Apparatus — sintered glass crucible No. 3 porosity.

3.3 Reagents

3.3.1 *Aqua Regia* — one part of concentrated nitric acid (sp gr 1·42) and three parts of concentrated hydrochloric acid (sp gr 1·16).

3.3.2 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962*).

3.3.3 *Sulphur Dioxide Solution* — saturated.

3.3.4 *Ammonium Chloride* — solid.

3.3.5 *Ammonium Chloride Solution* — 38 percent (*w/v*).

3.3.6 *Dimethylglyoxime Solution* — one percent (*w/v*) in water.

3.4 Procedure

3.4.1 Transfer 1·000 g of the metal to a 250-ml beaker and dissolve in 50 ml of aqua regia, heating on the hot-plate, if necessary. When solution is complete wash down the sides of the beaker and evaporate the solution to a very small volume. Take up with a small quantity of water and, if there is any precipitate, dilute to 150 ml and allow to stand on a hot-plate for two hours. Filter the solution and wash with hot water acidulated with concentrated hydrochloric acid.

3.4.2 Evaporate the solution nearly to dryness three times, using 20 ml of concentrated hydrochloric acid each time. Dilute the solution to 200 ml with hot water and add 10 ml of sulphur dioxide solution with stirring and allow to stand for a few minutes. If any precipitate appears add another 5 ml of sulphur dioxide solution and allow to settle on a hot-plate for a few hours.

3.4.3 Filter off the solution and wash the precipitate with hot water containing a little concentrated hydrochloric acid. Evaporate the filtrate to a small volume. Add ammonium chloride to the cold solution until just saturated. Dissolve any undissolved ammonium chloride with a few drops of concentrated hydrochloric acid. Allow to stand for a few hours, filter off the precipitate and wash it with ammonium chloride solution.

*Specification for hydrochloric acid (*revised*).

3.4.4 Dilute the filtrate to 250 ml in a volumetric flask and take an aliquot of 20 to 25 ml. Dilute to 200 ml and add 10 ml of concentrated hydrochloric acid. Add a slight excess of boiling solution of dimethylglyoxime to the cold solution to precipitate palladium (0·1 g of palladium requires 0·22 g of the reagent). Allow the solution to stand for an hour and filter through a weighed sintered glass crucible No. 3 porosity. Wash the dimethylglyoximate precipitate with concentrated hydrochloric acid and then with hot water. Dry at 110°C to constant weight.

3.5 Calculation

$$\text{Palladium, percent} = \frac{A \times 31{\cdot}67}{B}$$

where

A = weight in g of the palladium dimethylglyoximate precipitate, and

B = weight in g of sample represented by the aliquot.

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